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Secondary organic aerosol formation from the oxidation of a mixture of organic gases in a chamber

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1. Introduction

Particles suspended in the air can constitute a potential risk for human health and ecosystems (Pope and Dockery, 2006), specially the finest fraction. Although PM₁₀ (particles with a maximum diameter of 10 μm) have been included in European directives for a longer time (Directive 1999/30/EC) air quality objectives for finer particles have been just very recently established. For particles with a mean diameter lower than 2.5 μm (PM_{2.5}) the UE Directive 2008/50/EC has set a 25 $\mu\text{g}/\text{m}^3$ threshold for the annual mean concentration.

Although the term aerosol includes the particles and the gas in which they are suspended, commonly both terms, particles and aerosols, refer to particles in the atmosphere. A variety of inorganic and organic chemical compounds can be present in the particulate phase. The organic fraction can account for a 20 - 90 % of the finest fraction, according to some authors, such as (Kanakidou et al., 2005) and, therefore, the knowledge of this fraction is important to prevent human health risks. Both inorganic and organic aerosols can be directly emitted (primary aerosols) or can be formed in the atmosphere as a consequence of multiple physical and chemical processes (secondary aerosols). The presence of secondary organic aerosols (SOA) is specially relevant in urban areas (Zhang et al., 2007).

SOA is mainly produced from the oxidation of volatile organic compounds (VOCs), whose products present a sufficiently low volatility to partition into the particle phase according to the gas-particle partitioning theory (Odum et al., 1996) and then nucleate and grow to form organic particles. Presently, SOA is thought to be mainly constituted by polymers, formed through particle phase heterogeneous reactions (Kalberer et al., 2004). Other main components include organic nitrates, such as peroxy nitrates and peroxyacyl nitrates (Camredon et al., 2007; Kroll and Seinfeld, 2008), and carboxylic acids (Barsanti and Pankow, 2006). In spite of the fact that SOA formation has been the focus of many recent studies, some aspects continue to be not well understood. Simulation chambers represent an ideal vehicle to evaluate SOA formation potential by emitting selected VOCs in the presence of an oxidant under controlled conditions. Many studies in chambers have contributed to increase the knowledge of the oxidation processes of individual organic gases or simple mixes of them. VOCs related to anthropogenic emissions, such as substituted aromatics

(trimethylbenzenes, xylenes and toluene) and alkanes contained in gasolines, are potential SOA precursors in city areas, and they have been thoroughly studied in chamber experiments. Also VOCs related to biogenic emissions, such as isoprene and terpenes (limonene and pinenes) have been widely studied in chambers, as their contribution to global SOA formation is notorious (Claeys et al., 2004; Kleindienst et al., 2006; Leungsakul et al., 2005). OH-initiated is the most common oxidation pathway (Healy et al., 2008; Hu et al., 2007; Lim and Ziemann, 2005; Song et al., 2005; Weitkamp et al., 2007) and thus most of the studies in chambers have been focused on the reaction of the previously mentioned VOCs with this radical.

Recent publications suggest, however, that more complex VOCs mixtures should be used in chamber experiments in order to achieve a more realistic picture of the oxidative processes taking place in real polluted atmospheres (Hallquist et al., 2009). In this chapter SOA formation from a mixture of 1,3,5-TMB (1,3,5-trimethylbenzene), toluene, o-xylene and octane in the presence of an oxidant (nitrous acid, HONO) is evaluated at a 20% of relative humidity. For this purpose, a comprehensive gas phase chemistry and aerosol characterization is presented.

2. Experimental

The experiment was carried out in the EUPHORE facility located in CEAM (Valencia, Spain), a half-spherical Teflon outdoor chamber that allows the transmission of more than 80% of sunlight. Figure 1 illustrates the chamber when it is closed (left side of the picture), when it is being opened (central picture) and opened to sunlight (right side of the figure). The EUPHORE facility has been described in detail somewhere else (Becker, 1996; Volkamer et al., 2001).



Fig. 1. EUPHORE Photoreactor: closed (left side of the figure), while opening (middle of the figure), and opened to sunlight (right side of the figure).

Several analytical equipments provided information of some physical variables (temperature, radiation, humidity, pressure) and chemical concentration of many inorganic and organic gas compounds. Multiple measurement techniques, such as Gas Chromatography coupled with Mass Spectrometer (GC-MS), Fourier Transform Infrared Spectrometry (FTIR), High Pressure Liquid Chromatography (HPLC), Gas Chromatography (GC-ECD and GC-FID/PID), Absorptive Sampling Solid Phase Microextraction (SPME) were used to monitor the gas concentration of reactants and products.

Regarding the particle phase, aerosol concentration was monitored in an on-line way with a TEOM (Tapped Element Oscillating Monitor) and a SMPS (Scanning Mobility Particle Sizer).

This latter provides also information about the diameter particle distribution by classifying the aerosol particles by their electrical mobility. Also, three low volume samplings were taken during the experiment and one high volume once the chamber was closed, in order to analyze aerosol composition via gas chromatography and ion chromatography.

The experiment described in this chapter was performed on June, 17th, 2008, as a part of the campaign performed by the authors in 2008 described in recent publications (Vivanco et al., 2010). Experimental conditions are summarized in Table 1. A mixture of volatile organic compounds and HONO was introduced into the chamber.

	Time		Concentration (ppb)
Parents VOCs intro	7:11	1,3,5-TMB	151
HONO introduction	8:01	Toluene	99
Water introduction	8:48	o-xylene	17
Opening	10:33	Octane	80
Closure	15:23	HONO	98
		Relative Humidity	20%

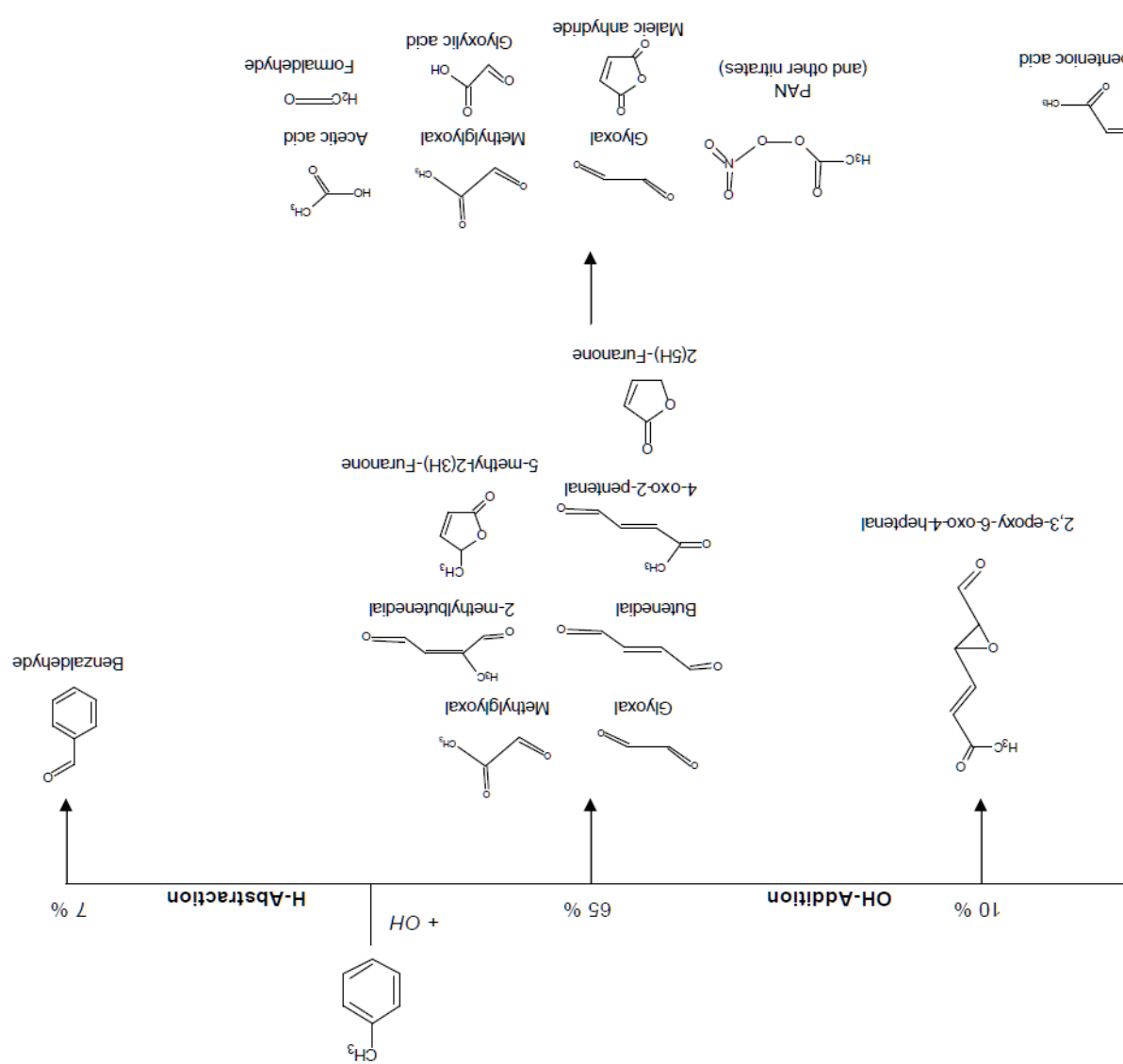
Table 1. Experimental conditions

After the parent VOCs the oxidant was introduced. Also, humidity conditions were prepared by introducing pulverized water into the chamber. Then, the chamber was opened to the sunlight.

3. Gas phase chemistry of the parent VOCs

In this section, a study about the atmospheric photochemical reactions is done, focusing on the oxidation pathways of the parent VOCs. These pathways consist in multiple oxidation steps which lead to the formation of multiple compounds. A very useful source of knowledge for atmospheric oxidation pathways is the Master Chemical Mechansim, developed by the University of Leeds (Jenkin et al., 2003; Saunders et al., 2002). The latest version of this mechanism, MCM v3.1 (Bloss et al., 2005), takes into account most of the kinetic and mechanistic data available to date.

The atmospheric oxidation of a certain compound is conditioned by its own structure and by the nature of the initial oxidant. Nitrous acid (HONO) was used as the oxidant compound and therefore, the major initial oxidant is the OH radical, formed by HONO photolysis. The reaction of the aromatic VOCs emitted in this experiment with the OH radical have been previously studied by several authors (Atkinson and Arey, 2003; Bloss et al., 2005; Hamilton et al., 2005; Huang et al., 2006; Johnson et al., 2004; Wagner et al., 2002). Two main reaction pathways can be identified in the oxidation of toluene, o-xylene or 1,3,5-TMB with OH: H-abstraction and OH-addition. The H-abstraction is considered as the minor route and leads to the formation of aromatic aldehydes. The OH-addition can occur in three differet ways: through the phenolic, the epoxy-oxy and the peroxy-bicyclic routes. The phenolic and epoxy-oxy routes lead to the formation of phenolic and epoxyde compounds respectively, while the peroxy-bicyclic route produces the opening of the aromatic rings and the formation of oxygenated products, which may lead to the formation of SOA if their volatility is low enough. This last route is considered as the major oxidation pathway according to the reactions included in the MCM v.3.1. A scheme of the oxidation pathways for toluene, o-xylene and 1,3,5-TMB is presented in figures 2a, 2b and 2c.



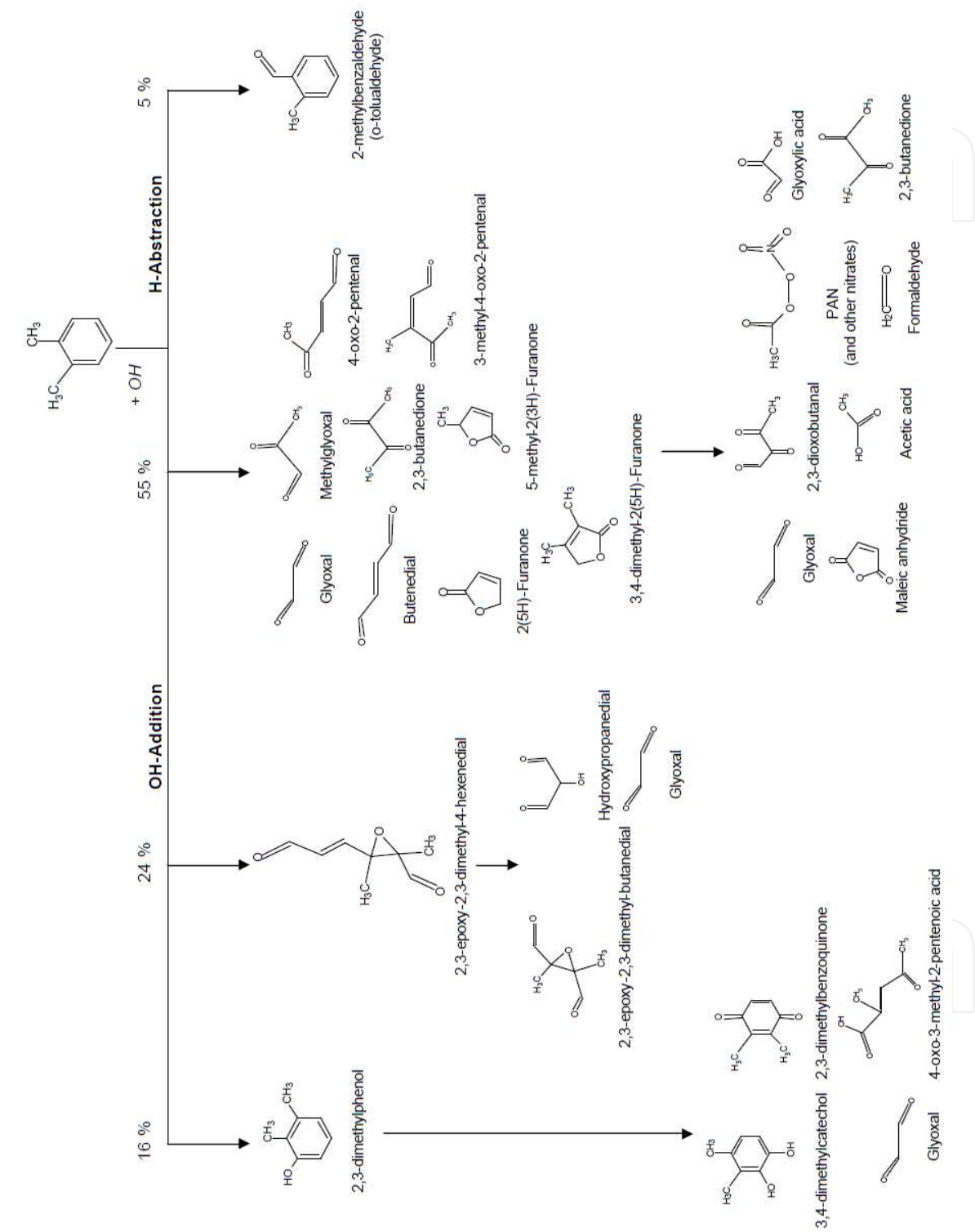


Fig. 2b O-xylene oxidation pathways scheme (MCM v.3.1)

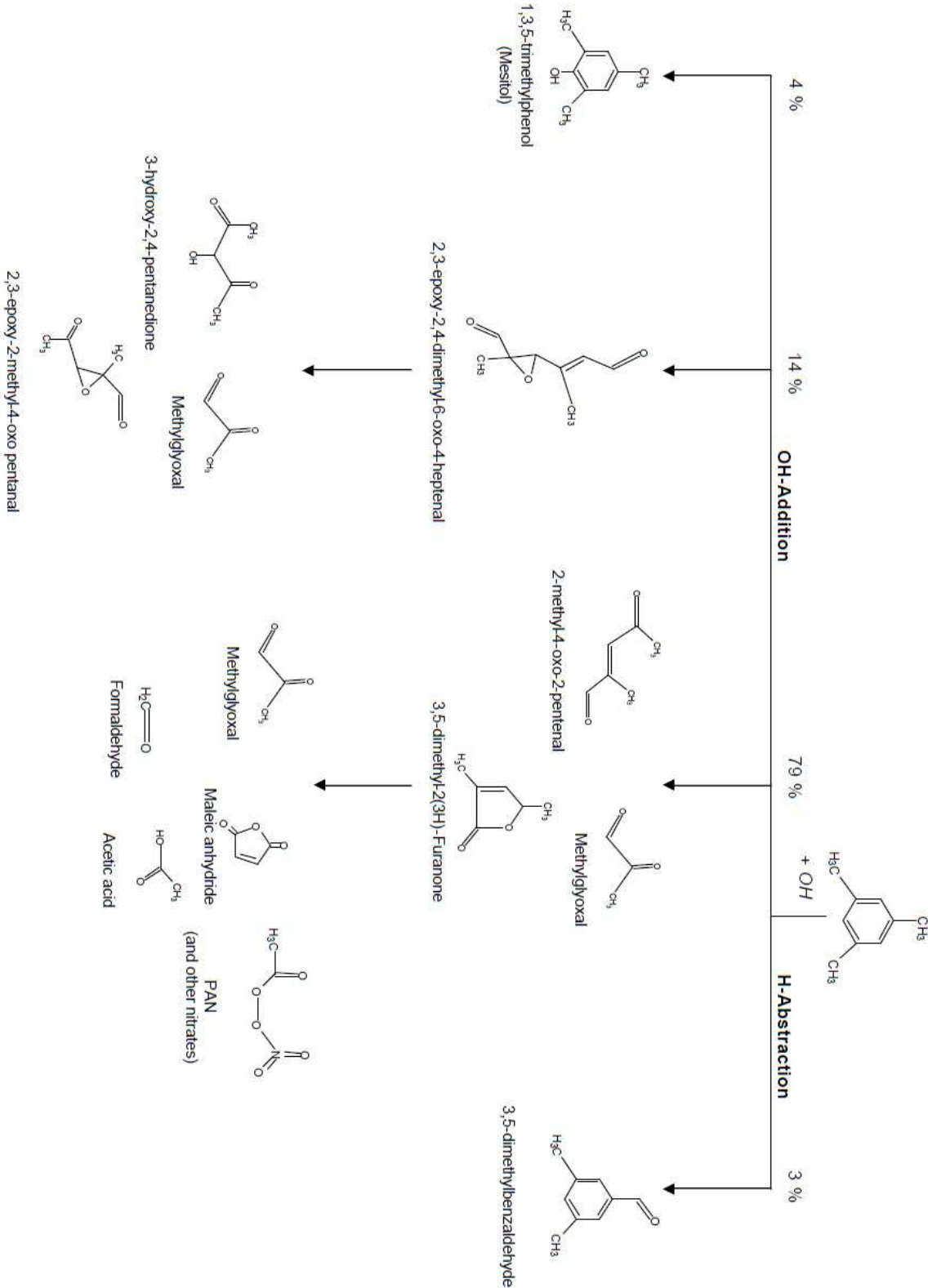


Fig. 2c. 1,3,5-TMB oxidation pathways scheme (MCM v.3.1)

In the case of octane, as for the rest of alkanes, the main oxidation pathway is the H-abstraction (Jordan et al., 2008; Lim and Ziemann, 2005). Figure 3 shows the main products formed during the octane oxidation, based on the reactions included in the MCM v.3.1:

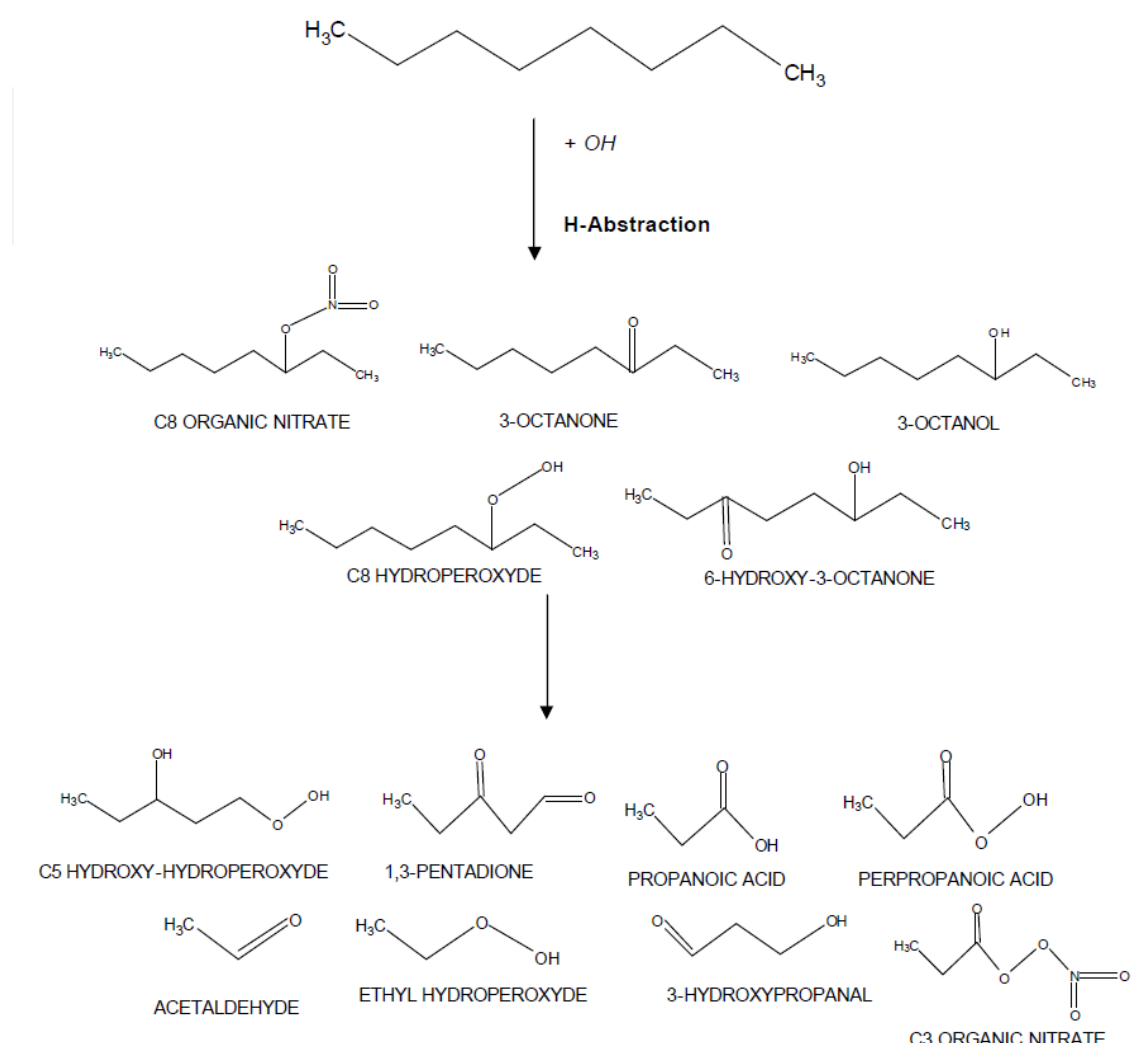


Fig. 3. Octane oxidation pathway scheme (based on MCM v3.1)

4. Results for the gas phase

Once the chamber is opened to the sunlight, the oxidation of the mixture of VOCs starts by reacting with the OH radical, formed from the photolysis of HONO:



OH radical is responsible for the initial oxidation of the VOCs by both OH-addition and H-abstraction. Although not presented in figures 3 and 4, an intermediate acyl peroxy radical is formed (RO₂), which may undergo several instantaneous reactions to form the resulting oxidation products.

Figure 4 illustrates a scheme of the overall processes expected to take place inside the chamber. Once the light enters the chamber new gas products and particles are formed due to oxidation processes occurring in both gas and particle phases.

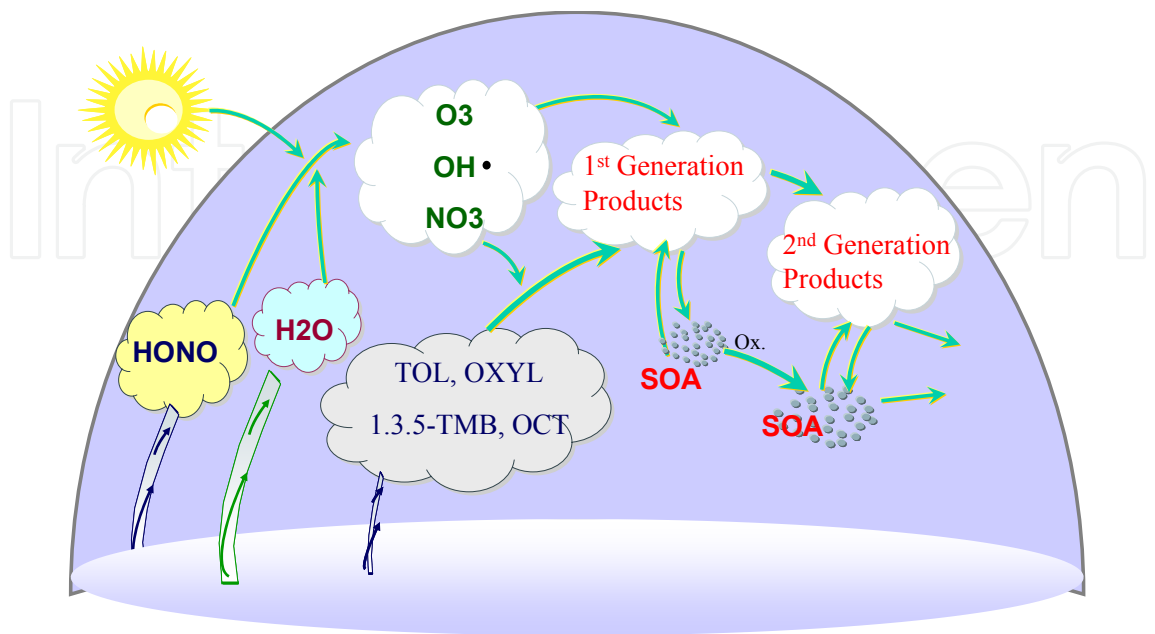


Fig. 4 Illustration of processes expected to take place during the experiment

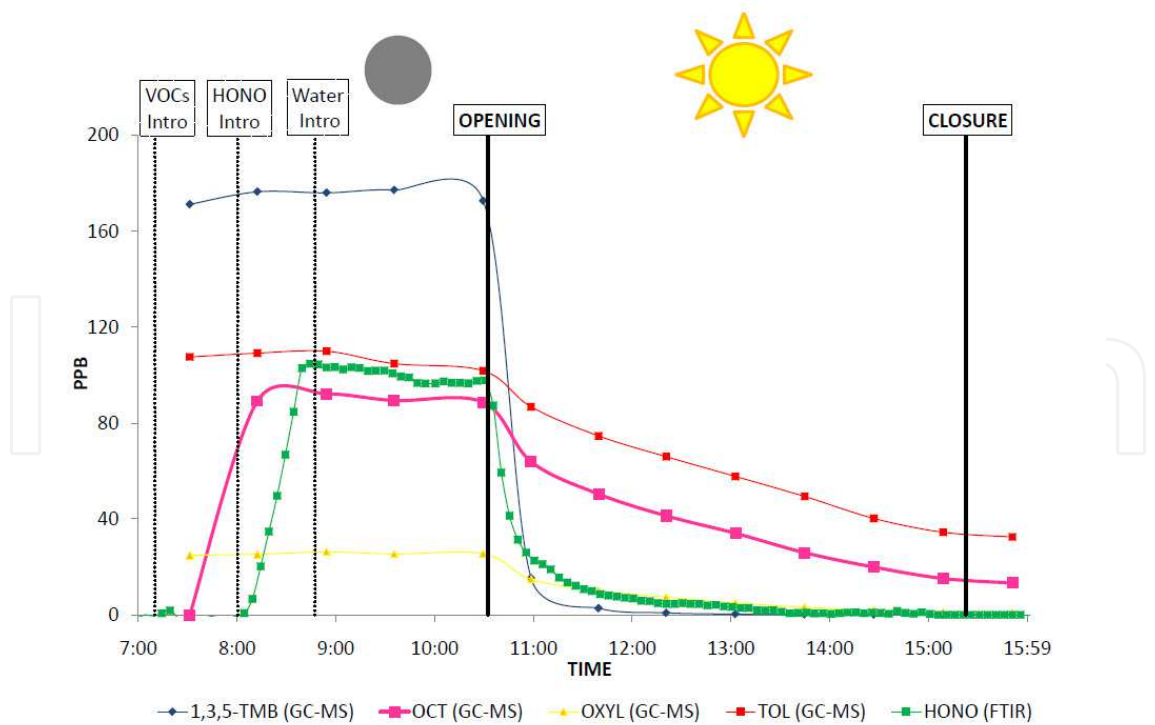


Fig. 5. Time series showing HONO, toluene (TOL), o-xylene (OXYL), 1,3,5-TMB and octane (OCT) concentration

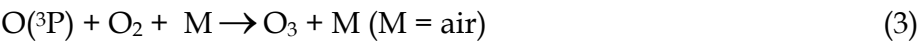
Time series showing the concentration of the initial reactants (the mixture of VOCs and HONO) are shown in Figure 5. The immediate and pronounced decay of HONO concentration is clearly observed when light enters the chamber (green line).

Also, a very strong concentration decrease is observed for 1,3,5-TMB (blue line). This fact is related to the highest reactivity of this compound with the OH radical, compared to the other three organic gases. Table 2 includes the OH-reactivity constant for the four gases.

COMPOUND	kOH (10 ¹² moles/cm ³ .s)
Toluene	5.74
o-xylene	13.6
Octane	8.61
1,3,5-TMB	56.7

Table 2. OH-reactivity constants for each parent VOC at 25° C (as given by MCM 3.1)

Ozone is a major product from the oxidation processes. In a clean atmosphere, there is a photoequilibrium between NO, NO₂ and O₃ and therefore no net ozone is produced (Atkinson, 2000):



However, in the presence of VOCs, this equilibrium is broken due to reactions of NO with RO₂ and HO₂ radicals formed during the oxidation of VOCs:



consuming NO but not ozone and, therefore, leading to a net production of ozone, a well known atmospheric pollutant. Figure 6 shows the increasing ozone concentration and the strong decrease of 1,3,5-TMB concentration produced when the chamber is opened. This sudden growth of ozone concentration is clearly related to the broken equilibrium described above.

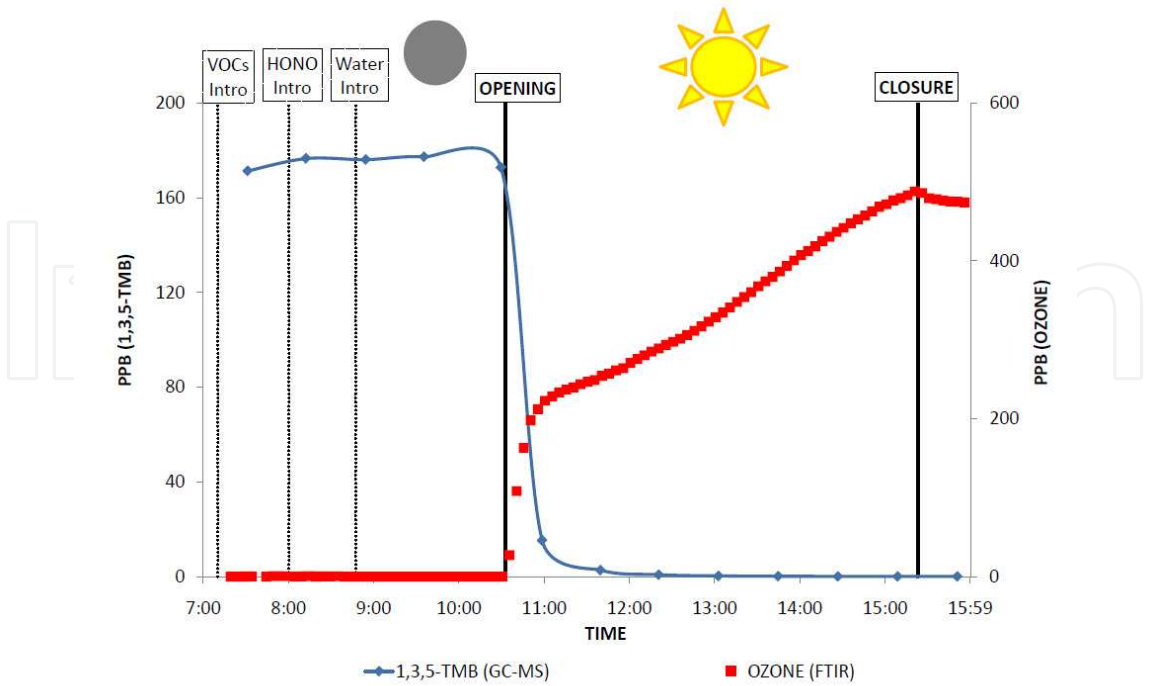


Fig. 6. Time series showing ozone and 1,3,5-TMB concentration

Besides ozone, a great variety of products were also identified during the experiment. Figure 7 shows the temporal evolution of the major products concentration. The parent VOCs and HONO have been also included in the figure in order to give a complete picture of the formation and decay times during the whole experiment.

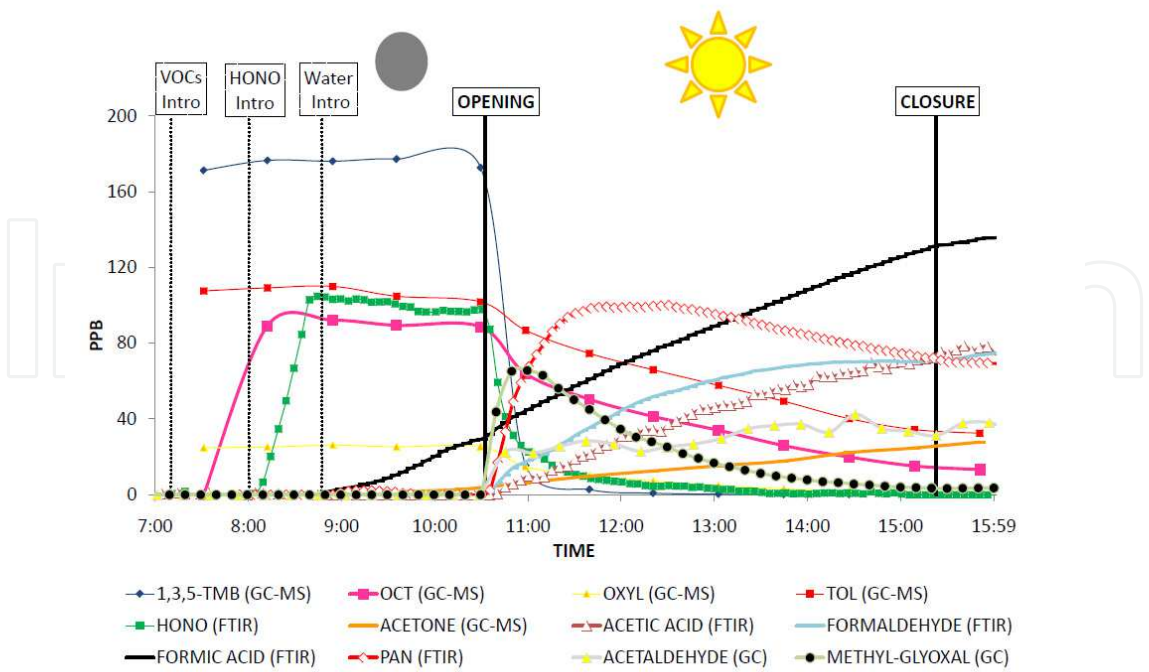


Fig. 7. Major products identified during the experiment (parent VOCs and HONO also included).

Peroxyacetyl nitrate (PAN) is one of main products formed inside the chamber. This nitrate is produced through the reaction of an acyl peroxy radical (RO₂) with NO₂:



and has a medium lifetime of 30 minutes, being thermal decomposition its major loss process at lower altitudes (Talukdar et al., 1995). PAN can partition to the particle phase and it has been previously identified as an important SOA constituent (Bonn et al., 2004; Johnson et al., 2004).

Methylglyoxal (2-oxopropanal) is a well known product from toluene, o-xylene and 1,3,5-TMB oxidation (Healy et al., 2008; Jang and Kamens, 2001; Volkamer et al., 2001). This dialdehyde can further react to form smaller compounds such as methanol, formadehyde, acetic acid and it can also produce PAN. In addition, methylglyoxal can partition into the particle phase. It has been reported that it can undergo accretion reactions (non-oxidative oligomer formation) to form hemiacetals due to the hydration of its aldehyde groups (Barsanti and Pankow, 2005; Loeffler et al., 2006). As a consequence of these processes, methylglyoxal presents an intermediate product concentration profile, with a clearly visible maximum peak.

Some other simple carbonyl products such as acetone, formaldehyde and acetaldehyde were also identified. In the case of formaldehyde, it can be produced from the oxidation of aromatic VOCs products (glyoxal, methylglyoxal, 2,3-butanedione or (5H)-2-furanone). Acetaldehyde can be mainly formed from the reaction of 3-octanone (an octane oxidation product) with OH radical. Acetone, however, is mainly formed from the ozonolysis of 3-methyl-4-oxo-2-pentenal, an o-xylene oxidation product. Ozonolysis reaction rates are very low (for a given compound, O₃-reactivity constants are generally several orders of magnitude lower than OH-reactivity constants), so little quantities of acetone are produced in the experiment, as it can be seen in Figure 7.

Formic and acetic acids can be formed in the chamber from the aqueous phase oxidation of their respective aldehydes (Chebbi and Carlier, 1996) and, in the case of acetaldehyde, also from the oxidation of aromatic VOCs oxidation products such as methylglyoxal, 2,3-butanedione and 3-methyl-4-oxo-2-pentenal. It has also been reported that formaldehyde reaction with hydroperoxy radicals HO₂ can be a significant source of formic acid in the gas phase (Khwaja, 1995). However, the most remarkable aspect about the formic acid is that, as it can be seen in Figure 7, it starts to be formed before the opening of the chamber. The formation of this acid coincides with the introduction of water in the chamber, suggesting that there is an additional formic acid formation way that does not include a photochemical activation.

In addition to the products presented in Figure 7, some other compounds in much lower concentrations were identified (Figure 8).

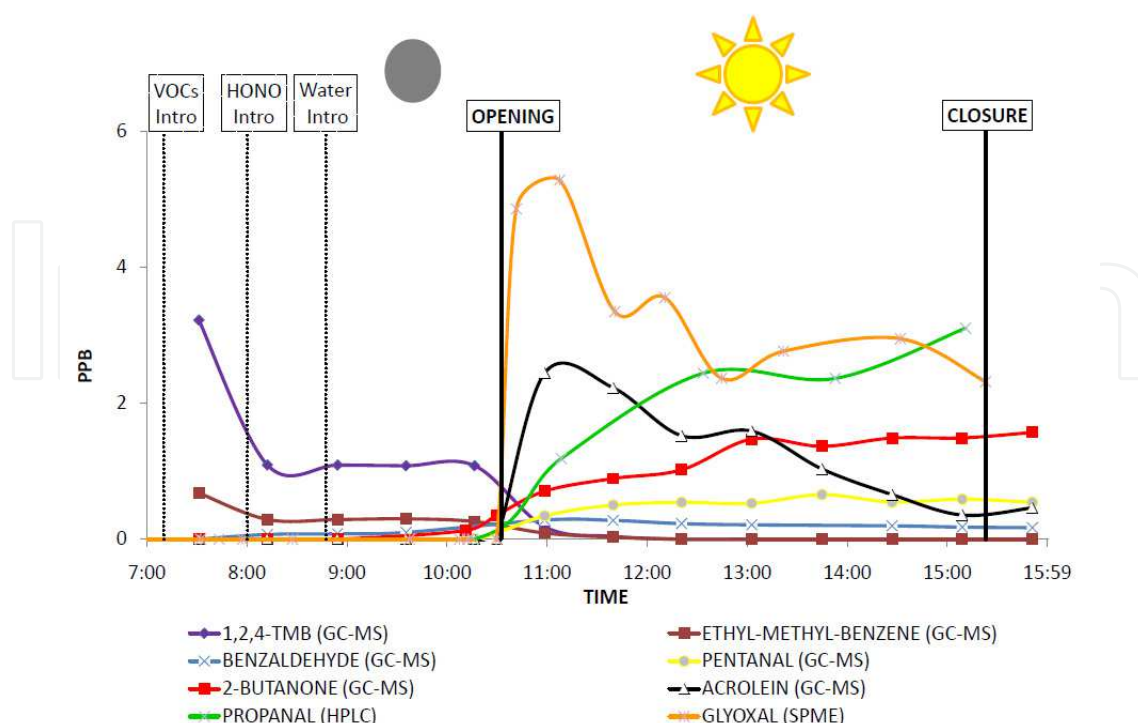


Fig. 8. Time series showing the concentration of some trace products

1,2,4-TMB and ethyl-methylbenzene (also known as ethyl-toluene) entered the chamber with the parent VOCs mixture in trace concentrations, while the presence of benzaldehyde in the chamber means that the H-abstraction pathway takes place at least for toluene, as benzaldehyde is its corresponding aromaldehyde. This is in concordance with the relative branching ratios predicted by MCM v.3.1 for the three gases, as toluene has the highest one (7 %) for the H-Abstraction route.

Glyoxal is a ring opening oxidation product from toluene and o-xylene (Volkamer et al., 2001). In the same way as methylglyoxal, this compound presents a high water solubility and can partition into the particle phase and form oligomers (Hastings et al., 2005; Hu et al., 2007; Volkamer et al., 2007). This fact could explain the low glyoxal gas phase concentration found in the experiment.

The small concentrations of acrolein, 2-butanone (butanone), propanal and pentanal measured through the experiment indicate that those are minor oxidation products from the parent VOCs.

5. Aerosol phase

The objective of the experiment was to determine the secondary organic formation from the mixture of the selected VOCs. As no aerosol was emitted all the aerosols recorded in the chamber have a secondary origin. Not only organic particles can be formed, but also some inorganic salts can be potential products of the reactant system. To identify these salts, ionic chromatography was applied. Figure 9 shows nitrates and sulfates contribution for the four samplings taken during the experiment (left side of the figure), as well as the characterization of the resulting organic mass (right side of the figure).

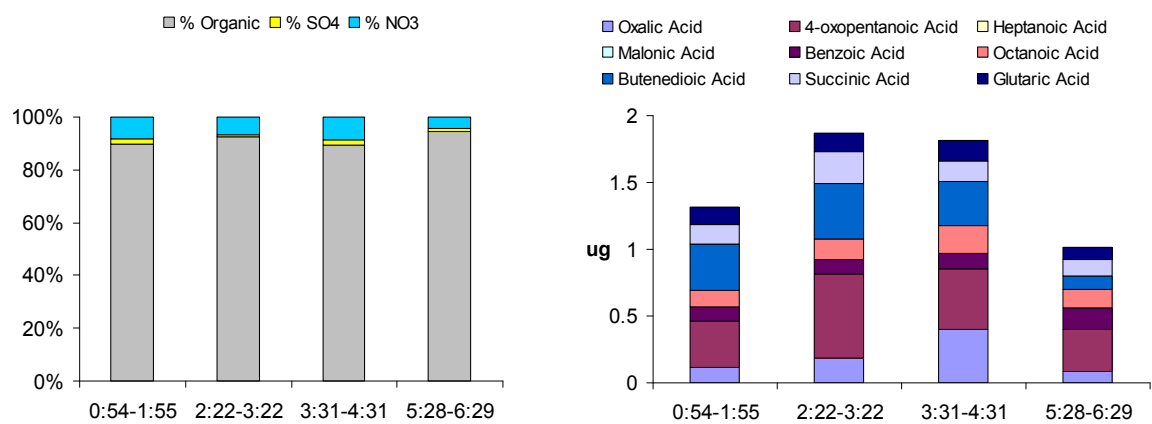


Fig. 9. Inorganic (left side) and organic (right side) filter characterization. The sampling time of each filter is presented in the x axis (time zero represents the opening of the chamber).

It can be seen that the inorganic contribution to the total aerosol mass is very low during the experiment. The small sulfate amount is similar to that found in blank filters. Nitrates can be formed due to the heterogeneous reaction of NO_2 with the water drops stuck on the chamber walls, driving to HNO_3 formation and, eventually, nitrates. Only a minimum quantity of the organic mass (about 60 – 90 μg in the first three filters and about 250 μg in the fourth) was identified, in a similar way to previous studies (Hamilton et al., 2005; Sato et al., 2007). Most of the acids identified were already detected in previous studies (Baltensperger et al., 2005; Hamilton et al., 2005; Jang and Kamens, 2001; Sato et al., 2007).

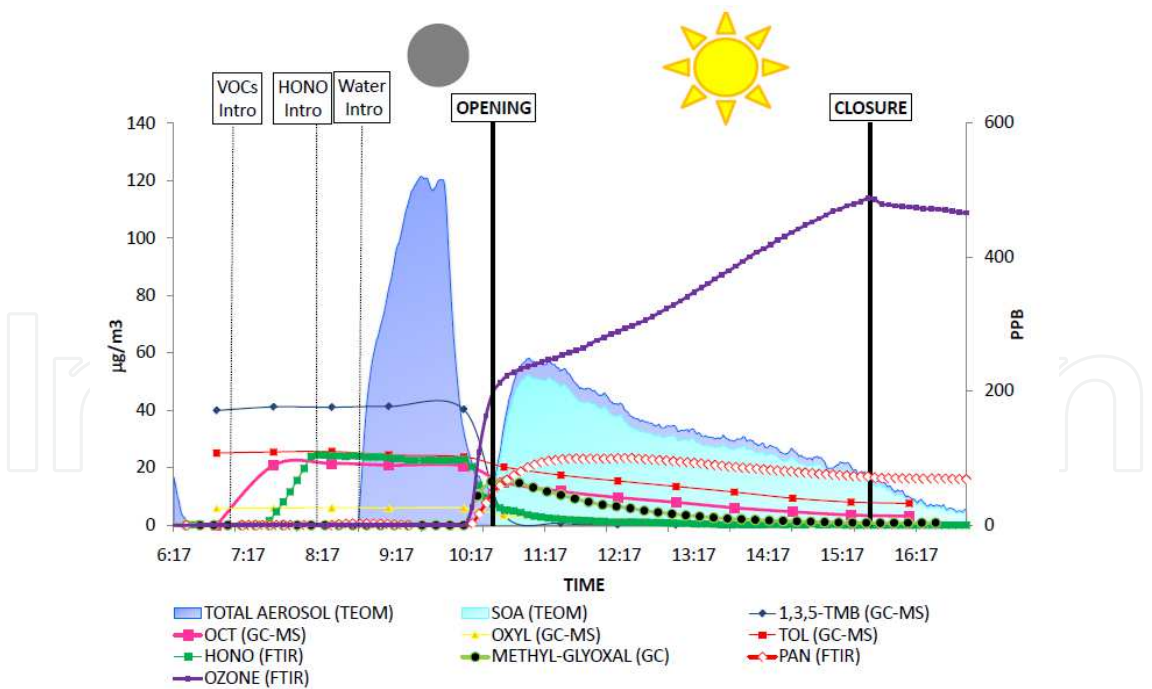


Fig. 10. Time series showing aerosol concentration measured with the TEOM (shaded blue area) and some other gases concentration. Aerosol concentration measured with TEOM is presented in Figure 10 (dark blue area). Particles start to be formed once the chamber is opened. Inorganic contribution estimated

from the filters was discounted from the total aerosol concentration in order to take an idea of the organic content (SOA, light blue area in Figure 10). The particles formed before the opening of the chamber correspond to small drops of water that are introduced into the chamber to create the 20% of relative humidity conditions. Scale for gases is presented in the right y-axis (ppb) while particle concentration is presented in the left one, in $\mu\text{g}/\text{m}^3$.

While other gas products such as ozone present a continuously increasing behaviour, particles are mainly formed during the first hour of the experiment. The initial formed particles present a small diameter and start growing by coagulation processes due to collisions between them (Kulmala et al., 2004).

The results provided by SMPS regarding particle size are presented in Figure 11. They reveal a growth of the aerosols. It is important to notice that the formation of detectable particles ($> 17 \text{ nm}$) starts approximately ten minutes after the opening of the chamber (purple band at 10:42). Because of the detection limit of SMPS, no smaller particles can be detected and therefore initial particle formation due to nucleation can not be monitored. For this reason, this analysis focuses on the particle growth once the first particles are formed.

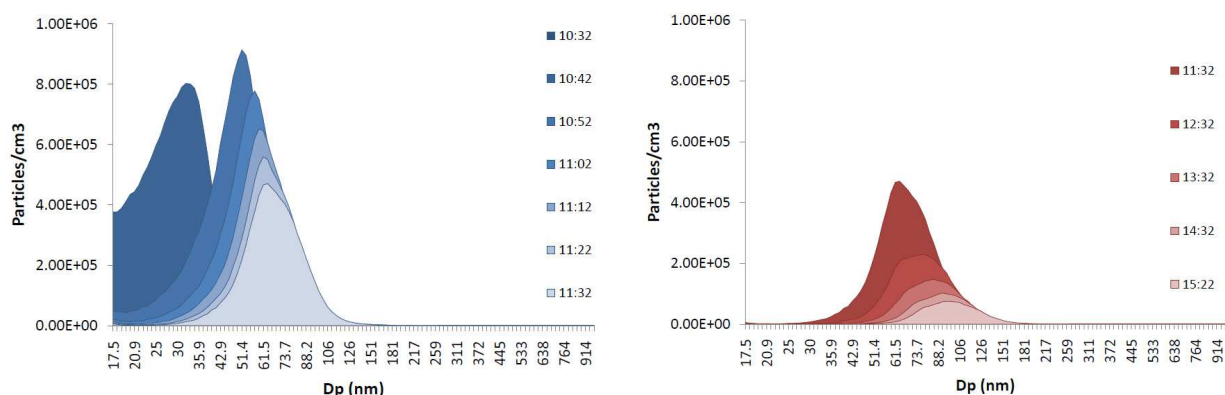


Fig. 11. Particle size distribution provided by SMPS

During the first hour after the opening of the chamber (left side of the figure) a quick growth in the particle diameter (D_p) takes place, coupled with a decrease in the number of particles, expressed as particles density (particles/ cm^3), which falls down from its maximum value ($9\text{E}+5$ particles/ cm^3). After this first hour, the particle diameter growth turns slower (right side of the figure). Coagulation and condensation of gas phase oxidation products can be the reason for this increase of the mean particle diameter (Sadezky et al., 2006). This increase in the mean particle diameter can be also inferred from Figure 12, where the temporal evolution of some selected diameters is presented.

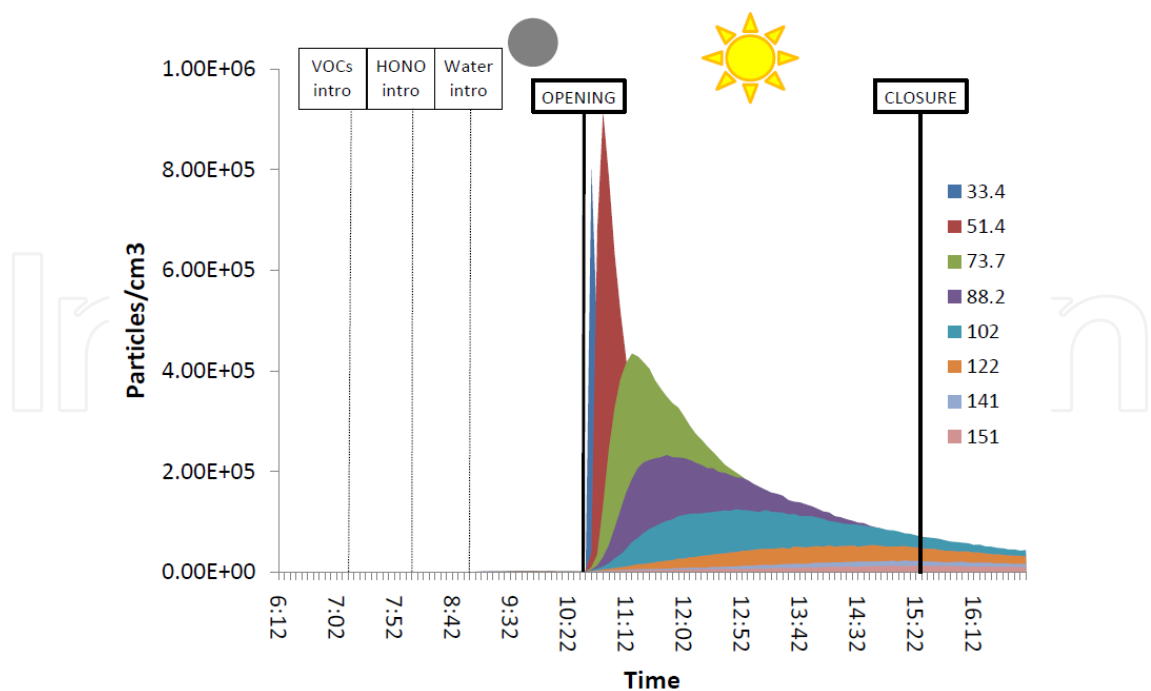


Fig. 12. Evolution of some selected D_p (nm) with time

The smallest particles are formed in high quantities at the beginning of the experiment (in the figure, diameters 33.4 nm and 51.4 nm) and then their concentration falls, while higher particles appear gradually, but in lower concentrations.

6. Conclusions and future challenges

In this chapter, a study focused on SOA formation from a mixture of anthropogenic VOCs is presented. 1,3,5-TMB resulted to be the most reactive VOC and therefore the initial steps of the photooxidation in the chamber are governed by its degradation. During the experiment, several organic compounds were measured and identified as products from specific oxidation pathways, some of them also known as relevant SOA constituents (PAN, methylglyoxal). The influence of the mixture of VOCs in ozone formation is also corroborated by a progressive concentration increase of this compound in the chamber.

Regarding the aerosol phase, maximum concentration is reached during the first hour after the opening of the chamber, indicating the formation of particles via nucleation of the condensed oxidation products. After this initial formation, the aerosol particles evolve and growth, possibly by coagulation processes and by the uptake to the particle phase of further oxidation products.

The chemical characterization revealed the presence of several carboxylic acids, but only a minor fraction of the total mass collected was identified. This limitation constitutes a common problem in chamber studies, as a consequence of current analytical techniques. Therefore, a more complete organic characterization represents a challenge and a necessity to better understand organic aerosols formation.

7. Acknowledgement

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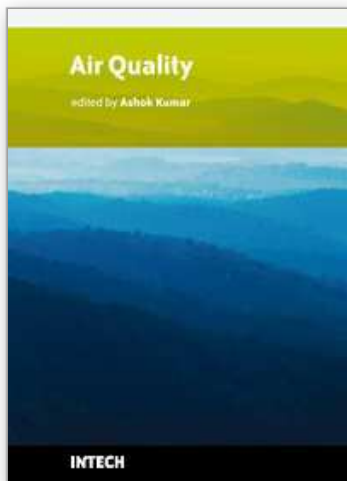
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Air pollution is about five decades or so old field and continues to be a global concern. Therefore, the governments around the world are involved in managing air quality in their countries for the welfare of their citizens. The management of air pollution involves understanding air pollution sources, monitoring of contaminants, modeling air quality, performing laboratory experiments, the use of satellite images for quantifying air quality levels, indoor air pollution, and elimination of contaminants through control. Research activities are being performed on every aspect of air pollution throughout the world, in order to respond to public concerns. The book is grouped in five different sections. Some topics are more detailed than others. The readers should be aware that multi-authored books have difficulty maintaining consistency. A reader will find, however, that each chapter is intellectually stimulating. Our goal was to provide current information and present a reasonable analysis of air quality data compiled by knowledgeable professionals in the field of air pollution.

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